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TITLE: High-temperature characterization of polymers

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PARENT-CASE:

This Application claims the benefit of Provisional
Application Ser. No.
60/080,652 filed Apr. 3, 1998. This application is
related to the following
U.S. patent applications filed on the date even herewith,
each of which is
hereby incorporated by reference for all purposes: Ser.
No. 09/285,393
entitled "Automatated Sampling Methods for Rapid
Characterization of Polymers",
filed Apr. 2, 1999 by Petro; Ser. No. 09/285,363 entitled
"Rapid
Characterization of Polymers", filed Apr. 2, 1999 by
Safir; Ser. No.
09/285,353 entitled "Flow-Injection Analysis and
Variable-Flow Light Scattering
Apparatus and Methods for Characterizing Polymers", filed
Apr. 2, 1999 by
Nielsen; and Ser. No. 09/285,393 entitled "Indirect
Calibration of Polymer
Characterization Systems", filed Apr. 2, 1999 by Petro.

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Brief Summary Text - BSTX:

Liquid chromatography is well known in the art for characterizing a polymer sample. Liquid chromatographic techniques employ separation of one or more components of a polymer sample from other components thereof by flow through a chromatographic column, followed by detection of the separated components with a flow-through detector. Approaches for liquid chromatography can vary, however, with respect to the basis of separation and with respect to the basis of detection. Gel permeation chromatography (GPC), a well-known form of size exclusion chromatography (SEC), is a frequently-employed chromatographic technique for polymer size determination. In GPC, the polymer sample is separated into components according to the hydrodynamic volume occupied by each component in solution. More specifically, a polymer sample is injected into a mobile phase of a liquid chromatography system and is passed through one or more chromatographic columns packed with porous beads. Molecules with relatively small hydrodynamic volumes diffuse into the pores of the beads and remain therein for longer periods, and therefore exit the column after molecules with relatively larger hydrodynamic volume. Hence, GPC can characterize one or more separated components of the polymer sample with respect to its effective hydrodynamic radius (R_h). Another chromatographic separation approach is illustrated by U.S. Pat. No. 5,334,310 to Frechet et al. and involves the use of a porous monolithic stationary-phase as a separation medium within the chromatographic column, combined with a mobile-phase composition gradient. (See also, Petro et al, Molded Monolithic Rod of Macroporous Poly(styrene-co-divinylbenzene) as a Separation Medium for HPLC Synthetic Polymers: "On-Column" Precipitation-Redissolution Chromatography

as an Alternative to Size Exclusion Chromatography of
 Styrene Oligomers and
 Polymers, Anal. Chem., 68, 315-321 (1996); and Petro et al,
 Immobilization of
 Trypsin onto "Molded" Macroporous Poly (Glycidyl
 Methacrylate-co-Ethylene
 Dimethacrylate) Rods and Use of the Conjugates as
 Bioreactors and for Affinity
 Chromatography, Biotechnology and Bioengineering, Vol. 49,
 pp. 355-363
 (1996)). Chromatography involving the porous monolith is
 reportedly based on a
 precipitation/redissolution phenomenon that separates the
 polymer according to
 size--with the precipitated polymer molecules selectively
 redissolving as the
 solvent composition is varied. The monolith provides the
 surface area and
 permeation properties needed for proper separation. Other
 separation
 approaches are also known in the art, including for
 example, normal-phase
 adsorption chromatography (with separation of polymer
 components being based on
 preferential adsorption between interactive functionalities
 of repeating units
 and an adsorbing stationary-phase) and reverse-phase
 chromatography (with
 separation of polymer components being based on hydrophobic
 interactions
 between a polymer and a non-polar stationary-phase). After
 separation, a
 detector can measure a property of the polymer or of a
 polymer component--from
 which one or more characterizing properties, such as
 molecular weight can be
 determined as a function of time. Specifically, a number
 of molecular-weight
 related parameters can be determined, including for
 example: the weight-average
 molecular weight ($M_{sub.w}$), the number-average molecular
 weight ($M_{sub.n}$), the
 molecular-weight distribution shape, and an index of the
 breadth of the
 molecular-weight distribution ($M_{sub.w} / M_{sub.n}$), known as
 the polydispersity
 index (PDI). Other characterizing properties, such as
 mass, particle size,

composition or conversion can likewise be determined.

Detailed Description Text - DETX:

The chromatographic column 102 further comprises a separation medium having a stationary-phase within the separation cavity. The separation medium can consist essentially of a stationary-phase or can also include, in addition thereto, an inert support for the stationary phase. The column 102 can also comprise one or more fillers, frits (for separation medium retention and/or for filtering), and various fittings and features appropriate for preparing and/or maintaining the column for its intended application. The particular separation medium to be employed as the stationary-phase is not critical, and will typically depend on the separation strategy for the particular chemistry of the polymer samples of interest, as well as on the desired detection, sample-throughput and/or information quality. Typical stationary-phase media can be a bed of packed beads, rods or other shaped-particles, or a monolithic medium (typically greater than about 5 mm in thickness), each of which can be characterized and optimized for a particular separation strategy with respect to the material, size, shape, pore size, pore size distribution, surface area, solvent regain, bed homogeneity (for packed shaped-particles), inertness, polarity, hydrophobicity, chemical stability, mechanical stability and solvent permeability, among other factors. Generally preferred stationary-phase include porous media (e.g., porous beads, porous monoliths), such as are suitable for gel permeation chromatography (GPC), and media suitable for precipitation-redissolution chromatography, adsorption chromatography, and/or reverse-phase chromatography. Non-porous particles or

empty columns and/or
capillaries with adsorptive walls can be used as well. If
beads are employed,
spherical beads are preferred over other shapes.
Particularly preferred
stationary-phase media for polymer characterization
applications are disclosed
in greater detail below, but can generally include silica,
cross-linked resins,
hydroxylated polyglycidyl methacrylates, (e.g.,
poly(2-3-dihydroxypropylmethacrylate)), poly(hydroxyethyl
methacrylate), and
polystyrenic polymers such as poly(styrene-divinylbenzene).